Acta Cryst. (1984). C**40**, 453–456

Basic Behaviour of Oxamide Dioxime: Structures of Di(oxamide dioximium) Squarate,* $2C_2H_7N_4O_2^+.C_4O_4^{2-}$ (I), and of Oxamide Dioximium Di(hydrogen squarate), $C_2H_8N_4O_2^{2+}.2C_4HO_4^-$ (II)

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(*Received* 12 *September* 1983; *accepted* 8 *November* 1983)

Abstract. Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, room temperature. (I) $M_r = 350{\cdot}26$, monoclinic, $P2_1/n$, $a = 3{\cdot}6797$ (9), $b = 21{\cdot}454$ (16), $c = 8{\cdot}807$ (4) Å, $\beta = 96{\cdot}93$ (3)°, $V = 690{\cdot}18$ Å^3, $Z = 2$, $D_x = 1{\cdot}68$ g cm^{-3}, $\mu = 1{\cdot}40$ cm^{-1}, $F(000) = 364$, $R = 0{\cdot}061$ for 394 observed independent reflections. In the $C_2H_7N_4O_2^+$ cation, present in the *s-trans* form, one oxime N atom is protonated. (II) $M_r = 346{\cdot}22$, monoclinic, $C2/c$, $a = 13{\cdot}248$ (5), $b = 6{\cdot}115$ (1), $c = 17{\cdot}918$ (6) Å, $\beta = 113{\cdot}69$ (3)°, $V = 1329{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}73$ g cm^{-3}, $\mu = 1{\cdot}48$ cm^{-1}, $F(000) = 712$, $R = 0{\cdot}040$ for 1540 observed independent reflections. The crystals contain mononegative $C_4HO_4^-$ anions and dipositive $C_2H_8N_4O_2^{2+}$ cations, protonated at both oxime N atoms, and adopting a *gauche conformation.*

Introduction. When 1,2-dione dioximes form bis-chelate complexes, they usually exhibit acidic properties. As is well known for bis(dimethylglyoximato)nickel(II), each ligand molecule loses one oxime proton, the other one forming an intramolecular H bridge. Basic behaviour of dioximes, on the other hand, is normally not considered. The base properties of oxamide dioxime (diaminoglyoxime, $oaoH_2$), however, have been investigated, as it was thought that the amino groups should be able to accept protons. Wenger, Monnier & Kapétanidis (1957) measured pK_a values and Pearse & Pflaum (1959) report the isolation of hydrochloric acid salts. The different preparative routes to $oaoH_2$ and the possible conformations of the molecule have been discussed (Ungnade, Kissinger, Narath & Barham, 1963). The structure determinations reported herein show that in both the monocation and the dication of $oaoH_2$ the protons are bound to the oxime N and not to the amino groups, and that *trans* and *gauche* forms can be obtained.

Experimental. The two title compounds were obtained as by-products when we investigated the reactivity of $[Ni(oaoH)_2]$ with squaric acid (Endres & Schendzielorz, 1983). $(oaoH_3)_2C_4O_4$ (I) was obtained when 2·5 mmol $[Ni(oaoH)_2]$ was added to a solution of 5 mmol squaric acid in 50 ml warm water. The resulting Ni complex was filtered off, and when the light blue-green filtrate was evaporated in air, long colourless columns of (I) crystallized together with the salt $[Ni(H_2O)_6]C_4O_4$. $(oaoH_4)(C_4HO_4)_2$ (II) formed when a slurry of 2·5 mmol $[Ni(oaoH)_2]$ in 50 ml water was dissolved by adding 2 ml concentrated HCl and this solution added to a solution of 5 mmol squaric acid in 20 ml water. The reaction mixture was heated to boiling, allowed to cool to room temperature, and filtered. On standing, large square orange-yellow crystals of (II) were obtained.

Syntex $R3$ diffractometer, monochromatic Mo $K\alpha$ radiation, lattice parameters from setting angles of 25 reflections, ω-scans background–peak–background. Two check reflections measured at intervals of 100 reflections, no absorption correction. Further details are in Table 1. Structures solved by direct methods, refinement based on F, $w = 1/\sigma^2(F)$, 'cascade matrix' least squares, *SHELXTL* (Sheldrick, 1981) on a Nova 3 computer, also used for plots. Scattering factors from *International Tables for X-ray Crystallography* (1974). Compound (I): Small number of data, hence only heaviest atoms (O) refined anisotropically. H atoms from difference Fourier map included but not refined,

Table 1. *Experimental details*

	$(oaoH_3)_2C_4O_4$ (I)	$(oaoH_4)(C_4HO_4)_2$ (II)
Crystal shape	Prism	Plate
Size (mm)	0·1 × 0·1 × 0·2	0·3 × 0·3 × 0·1
Max. 2θ (°)	50	60
Min. h, k, l	$0, 0, \overline{10}$	$0, 0, \overline{25}$
Max. h, k, l	4, 25, 10	18, 8, 25
Reflections measured	1391	2182
Reflections observed [$I > 2{\cdot}0\sigma(I)$]	466	1593
Unique observed reflections	394	1540
$R_{\text{int.}}$	0·038	0·021
Intensity variation of check reflections	±3·3%	±4·1%
R, R_w	0·061, 0·057	0·040, 0·038
S	2·05	2·26
$(\Delta/\sigma)_{\text{max}}$	0·001	0·18
Largest features in $\Delta\rho$ map	+0·34, −0·26 e Å^{-3}	+0·39 −0·18 e Å^{-3}

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* Squarate: anion of 3,4-dihydroxy-3-cyclobutene-1,2-dione. Oxamide dioximium is the mono- or dication of oxamide oxime.

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$U = 0{\cdot}06$ Å^2. Compound (II): Non-H atoms refined anisotropically, H atoms from difference Fourier map refined with individual isotropic temperature factors.

Discussion. Atomic coordinates are listed in Tables 2 and 3.* Bond distances and angles are in Tables 4 and 5. Fig. 1 shows the numbering scheme in (I); Fig. 2 shows an adjacent cation–anion pair of (II) and the numbering scheme. In (I) the centre of the squarate dianion coincides with a crystallographic inversion centre, and the $(oaoH_3)^+$ cation is in a general position. Its conformation is approximately *trans*; the torsion angle N(1)—C(3)—C(4)—N(4) is 32 (1)°. In (II) the hydrogen squarate anion is in a general position, whereas the $(oaoH_4)^{2+}$ dication is bisected by a crystallographic twofold axis perpendicular to the C(5)—C(5*a*) bond. The conformation is *gauche* with torsion angle N(1)—C(5)—C(5*a*)—N(1*a*) 42·4 (2)°. Within the relatively large standard deviations, the squarate dianion in (I) is really square. In the hydrogen squarate monoanion in (II), however, significant deviations from the square occur: The C—C bonds at C(1), 1·440 (2) and 1·444 (2) Å, are significantly shorter than those at C(3), 1·482 (2) and 1·481 (2) Å. Furthermore, the C(3)—O(3) distance, 1·238 (2) Å, is significantly shorter than the other C—O distances and close to the usual value for a C=O double bond. Thus the negative charge is probably delocalized between O(2) and O(4) *via* the chain O(2)—C(2)—C(1)—C(4)—O(4) where all the bonds have partial double-bond character. The squarate C—C bond distances in (I) seem longer than those in (II), which would be expected because of the increased charge, but the difference is hardly significant. In the crystals anions and cations are linked together by an extended network of H bridges. These are indicated in Figs. 3 and 4, which show stereoviews of the packing.

These structure determinations show that both *trans* and *gauche* forms of the oxamide cation can occur in solids, probably stabilized by the different H-bonding interactions. The monoprotonated cation together with squarate dianion is obtained from a solution containing squaric acid as the only acid. The presence of HCl in the preparation of (II) shifts the acid–base equilibrium to the doubly protonated cation and hydrogen squarate anion.

Neutral $oaoH_2$ has up to now always been found in the *trans* form when crystallizing together with $[M(oaoH)_2]$ complexes, M = Co, Ni, Cu (Endres, Genc & Nöthe, 1983). The additional protons in the cations are not attached to the amino but to the oxime N atoms, in contrast to earlier assumptions (Wenger *et al.*, 1957). This indicates that the lone electron pair on the oxime N is more basic than that of the amino N, consistent with the fact that in $oaoH_2$ complexes coordination occurs always *via* the oxime N atoms. In the older literature the possibility of coordination through the amino groups had been discussed, leaving the oxime groups free, *e.g.* in $[Ni(oaoH_2)_3]^{2+}$ (Tschugaeff & Surenjanz, 1907). X-ray structure determination, however, revealed that this mode of coordination does not take place (Endres & Jannack, 1980). Coordination through the amino groups in addition to that through the oxime groups may, however, occur, leading to the coordination polymers. This was observed in Co (Bekaroglu, Sarisaban, Koray & Ziegler, 1977) and in Cu complexes (Endres, Genc & Nöthe, 1983).

This work was supported by the Fonds der Chemischen Industrie.

Table 2. *Atomic coordinates* (×10^4) *and isotropic temperature factors* (Å^2 ×10^3) *for compound* (I), $(oaoH_3)_2C_4O_4$

	x	*y*	*z*	U/U_{eq}
C(1)	1161 (28)	435 (4)	5218 (10)	20 (2)
C(2)	−1244 (28)	161 (4)	3951 (11)	23 (3)
O(1)	2652 (18)	961 (3)	5534 (6)	25 (3)*
O(2)	−2711 (21)	354 (3)	2674 (7)	35 (3)*
C(3)	2911 (26)	3551 (4)	4271 (10)	19 (3)
C(4)	3489 (26)	2954 (4)	3474 (10)	22 (3)
N(1)	1481 (22)	4016 (4)	3395 (9)	29 (2)
N(2)	3763 (22)	3607 (3)	5767 (9)	30 (2)
N(3)	3016 (22)	2467 (4)	4300 (9)	29 (2)
N(4)	4275 (22)	2977 (4)	2021 (9)	30 (2)
O(3)	972 (25)	4563 (3)	4177 (8)	57 (3)*
O(4)	3628 (20)	1924 (3)	3415 (7)	41 (3)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3. *Atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 ×10^3) *for* $(oaoH_4)(C_4HO_4)_2$ (II)

$U_{eq} = \frac{1}{3}$ trace $\tilde{\mathbf{U}}$, $\tilde{\mathbf{U}}$ signifying the diagonalized $\mathbf{U}$ matrix.

	x	*y*	*z*	U_{eq}
C(1)	3695 (1)	4207 (2)	4932 (1)	23 (1)
C(2)	4340 (1)	3310 (2)	5725 (1)	22 (1)
C(3)	4097 (1)	1096 (2)	5359 (1)	22 (1)
C(4)	3429 (1)	2076 (2)	4555 (1)	23 (1)
O(1)	3399 (1)	6156 (2)	4624 (1)	36 (1)
O(2)	4867 (1)	4027 (2)	6429 (1)	28 (1)
O(3)	4344 (1)	−779 (2)	5632 (1)	32 (1)
O(4)	2863 (1)	1343 (2)	3864 (1)	32 (1)
C(5)	−613 (1)	5972 (2)	2372 (1)	19 (1)
N(1)	−1041 (1)	4183 (2)	2521 (1)	23 (1)
N(2)	−1174 (1)	7716 (2)	2025 (1)	26 (1)
O(5)	−2167 (1)	4159 (2)	2306 (1)	28 (1)

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* Lists of structure factors, anisotropic thermal parameters, H coordinates, and bond distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39014 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Bond distances* (Å) *and angles* (°) *in* $(oaoH_3)_2C_4O_4$ (I)

C(1)–C(2)	1·461 (13)	C(1)–O(1)	1·271 (11)
C(1)–C(2*a*)	1·471 (13)	C(2)–O(2)	1·257 (11)
C(2)–C(1*a*)	1·471 (13)	C(3)–C(4)	1·489 (13)
C(3)–N(1)	1·330 (12)	C(3)–N(2)	1·322 (11)
C(4)–N(3)	1·296 (12)	C(4)–N(4)	1·347 (12)
N(1)–O(3)	1·384 (10)	N(3)–O(4)	1·435 (10)
C(2)–C(1)–O(1)	136·9 (8)	C(2)–C(1)–C(2*a*)	90·3 (7)
O(1)–C(1)–C(2*a*)	132·9 (8)	C(1)–C(2)–O(2)	134·9 (8)
C(1)–C(2)–C(1*a*)	89·7 (7)	O(2)–C(2)–C(1*a*)	135·4 (8)
C(4)–C(3)–N(1)	116·2 (8)	C(4)–C(3)–N(2)	121·2 (8)
N(1)–C(3)–N(2)	122·7 (8)	C(3)–C(4)–N(3)	113·1 (8)
C(3)–C(4)–N(4)	118·3 (8)	N(3)–C(4)–N(4)	128·6 (9)
C(3)–N(1)–O(3)	114·5 (7)	C(4)–N(3)–O(4)	107·9 (7)

Table 5. *Bond distances* (Å) *and angles* (°) *in* $(oaoH_4)(C_4HO_4)_2$ (II)

C(1)–C(2)	1·440 (2)	C(1)–C(4)	1·444 (2)
C(1)–O(1)	1·305 (2)	C(2)–C(3)	1·482 (2)
C(2)–O(2)	1·251 (2)	C(3)–C(4)	1·481 (2)
C(3)–O(3)	1·238 (2)	C(4)–O(4)	1·245 (2)
C(5)–N(1)	1·308 (2)	C(5)–N(2)	1·306 (2)
C(5)–C(5*a*)	1·501 (3)	N(1)–O(5)	1·383 (2)
C(2)–C(1)–C(4)	93·1 (1)	C(2)–C(1)–O(1)	136·3 (1)
C(4)–C(1)–O(1)	130·6 (1)	C(1)–C(2)–C(3)	88·5 (1)
C(1)–C(2)–O(2)	137·0 (1)	C(3)–C(2)–O(2)	134·4 (1)
C(2)–C(3)–C(4)	89·9 (1)	C(2)–C(3)–O(3)	134·1 (1)
C(4)–C(3)–O(3)	135·9 (1)	C(1)–C(4)–C(3)	88·4 (1)
C(1)–C(4)–O(4)	136·6 (1)	C(3)–C(4)–O(4)	135·0 (1)
N(1)–C(5)–N(2)	124·8 (1)	N(1)–C(5)–C(5*a*)	116·2 (1)
N(2)–C(5)–C(5*a*)	119·0 (1)	C(5)–N(1)–O(5)	117·3 (1)

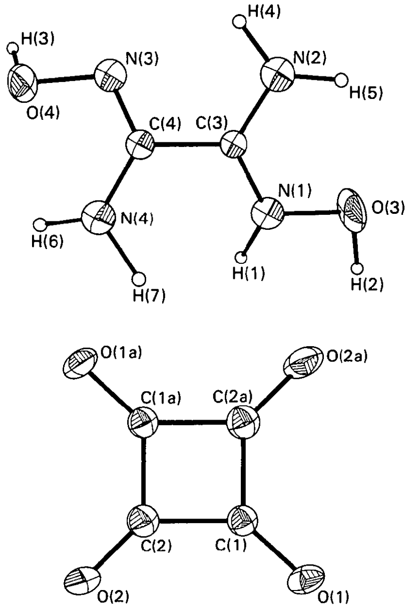

Fig. 1. The $C_2H_7N_4O_2^+$ cation and the $C_4O_4^{2-}$ anion in (I) with the numbering scheme. Thermal contours at 50% probability, H atoms with arbitrary radius.

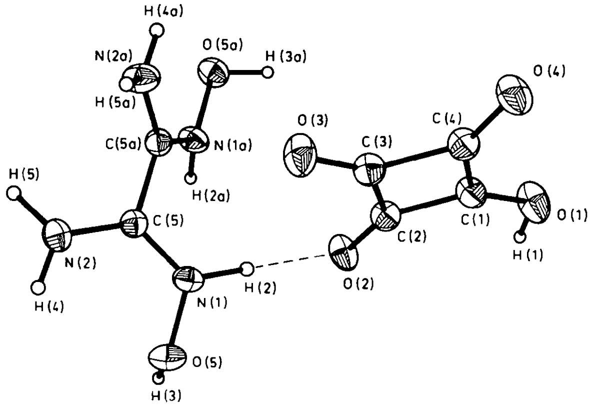

Fig. 2. View of an adjacent $C_2H_8N_4O_2^{2+}$-cation–$C_4HO_4^-$-anion pair in (II). Analogous to Fig. 1.

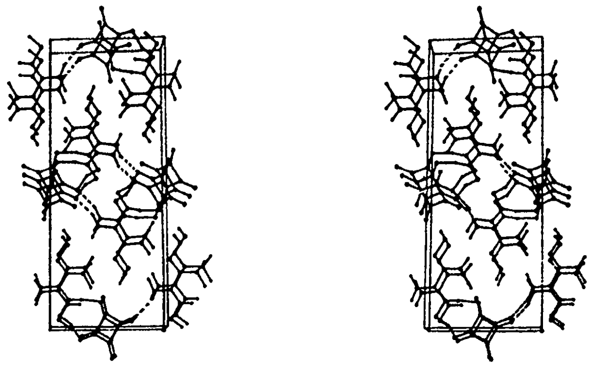

Fig. 3. Stereoview of the crystal packing in (I) with intermolecular H bridges indicated as dotted lines, viewed from a direction inclined at 10° from the *x* axis; *y* vertical, *z* horizontal.

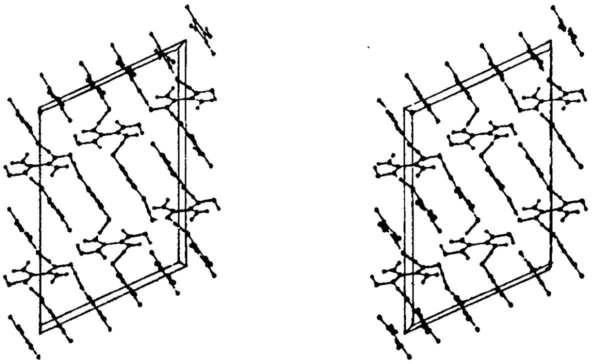

Fig. 4. Stereoview of the crystal packing in (II) with intermolecular H bridges, viewed along *y*; *x* horizontal, *z* vertical.

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Acta Cryst. (1984). C**40**, 456–464

Structures of Various Adducts Between 2,5-Dimethyl-1,4-benzoquinone or 2-Methyl-1,4-naphthoquinone and 1,1'-Bicycloalkenyls[*]

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(*Received* 13 *July* 1983; *accepted* 14 *November* 1983)

Abstract. (1) $C_{18}H_{22}O_2$, $M_r = 270\cdot37$, $P2_1/c$, $a = 8\cdot491\ (4)$, $b = 20\cdot029\ (10)$, $c = 8\cdot650\ (4)$ Å, $\beta = 96\cdot48\ (2)°$, $U = 1461\cdot67$ Å^3, $Z = 4$, $D_x = 1\cdot228$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot43$ cm^{-1}, $F(000) = 584$, $R = 0\cdot062$ for 1985 reflections. (2) $C_{20}H_{26}O_2$, $M_r = 298\cdot43$, $P2_1/c$, $a = 18\cdot509\ (9)$, $b = 9\cdot250\ (5)$, $c = 21\cdot229\ (10)$ Å, $\beta = 112\cdot56\ (3)°$, $U = 3356\cdot44$ Å^3, $Z = 8$, $D_x = 1\cdot181$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot40$ cm^{-1}, $F(000) = 1296$, $R = 0\cdot079$ for 3118 reflections. (3) $C_{19}H_{24}O_2$, $M_r = 284\cdot40$, $Pna2_1$, $a = 9\cdot080\ (5)$, $b = 15\cdot706\ (8)$, $c = 10\cdot994\ (6)$ Å, $U = 1567\cdot86$ Å^3, $Z = 4$, $D_x = 1\cdot205$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 5\cdot20$ cm^{-1}, $F(000) = 616$, $R = 0\cdot038$ for 1345 reflections. (4) $C_{22}H_{24}O_2$, $M_r = 320\cdot43$, $P2_12_12_1$, $a = 34\cdot069\ (15)$, $b = 7\cdot039\ (4)$, $c = 7\cdot093\ (4)$ Å, $U = 1700\cdot98$ Å^3, $Z = 4$, $D_x = 1\cdot251$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 5\cdot34$ cm^{-1}, $F(000) = 688$, $R = 0\cdot064$ for 1631 reflections. (5) $C_{21}H_{28}O_2$, $M_r = 312\cdot45$, $P2_12_12_1$, $a = 20\cdot091\ (10)$, $b = 9\cdot187\ (5)$, $c = 9\cdot331\ (5)$ Å, $U = 1722\cdot28$ Å^3, $Z = 4$, $D_x = 1\cdot205$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot41$ cm^{-1}, $F(000) = 680$, $R = 0\cdot070$ for 1230 reflections. (6) $C_{24}H_{28}O_2$, $M_r = 348\cdot49$, $P2_1$, $a = 19\cdot254\ (1)$, $b = 9\cdot841\ (5)$, $c = 9\cdot985\ (5)$ Å, $\beta = 93\cdot74\ (3)°$, $U = 1891\cdot83$ Å^3, $Z = 4$, $D_x = 1\cdot223$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 5\cdot13$ cm^{-1}, $F(000) = 752$, $R = 0\cdot057$ for 3145 reflections. (7) $C_{22}H_{30}O_2$, $M_r = 326\cdot48$, $P2_1/n$, $a = 18\cdot389\ (9)$, $b = 15\cdot398\ (8)$, $c = 6\cdot370\ (3)$ Å, $\beta = 90\cdot22\ (2)°$, $U = 1803\cdot67$ Å^{-3}, $Z = 4$, $D_x = 1\cdot202$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot40$ cm^{-1}, $F(000) = 712$, $R = 0\cdot061$ for 2191 reflections. (8) $C_{25}H_{30}O_2$, $M_r = 362\cdot51$, $P2_1/c$, $a = 16\cdot965\ (9)$, $b = 10\cdot122\ (5)$, $c = 12\cdot739\ (6)$ Å, $\beta = 111\cdot74\ (2)°$, $U = 2031\cdot94$ Å^3, $Z = 4$, $D_x = 1\cdot185$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot39$ cm^{-1}, $F(000) = 784$, $R = 0\cdot081$ for 1718 reflections. (9) $C_{22}H_{30}O_2$, $M_r = 326\cdot48$, $P\bar{1}$, $a = 15\cdot338\ (8)$, $b = 9\cdot268\ (5)$, $c = 7\cdot036$ Å, $\alpha = 80\cdot08\ (3)$, $\beta = 103\cdot41\ (3)$, $\gamma = 102\cdot70\ (3)°$, $U = 941\cdot33$ Å^3, $Z = 2$, $D_x = 1\cdot152$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot38$ cm^{-1}, $F(000) = 354$, $R = 0\cdot064$ for 2560 reflections. (10) $C_{22}H_{30}O_2$, $M_r = 326\cdot48$, $C2/c$, $a = 12\cdot513\ (6)$, $b = 14\cdot103\ (7)$, $c = 21\cdot044\ (11)$ Å, $\beta = 106\cdot03\ (3)°$, $U = 3569\cdot25$ Å^3, $Z = 8$, $D_x = 1\cdot215$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot41$ cm^{-1}, $F(000) = 1424$, $R = 0\cdot054$ for 2326 reflections. Crystal structures of eight adducts of 2,5-dimethyl-1,4-benzoquinone or 2-methyl-1,4-naphthoquinone and 1,1'-bicycloalkenyls containing rings of various sizes ranging from five- to seven-

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[*] (1) 6b,9-Dimethyl-1,2,3,4,5,6,6a,6b,10a,10b-decahydrobenz[*e*]indacene-7,10-dione.

(2) 8b,11-Dimethyl-1,2,3,4,5,6,7,8,8a,8b,12a,12b-dodecahydro-9,12-triphenylenedione.

(3) 7b,10-Dimethyl-2,3,4,5,6,7,7a,7b,11a,11b-decahydro-1*H*-cyclopenta[*l*]phenanthrene-8,11-dione.

(4) 7b-Methyl-2,3,4,5,6,7,7a,7b,13a,13b-decahydro-1*H*-benzo[*b*]cyclopenta[*l*]phenanthrene-8,13-dione.

(5) 4b,7-Dimethyl-2,3,4,4a,4b,8a,8b,9,10,11,12,13-decahydro-1*H*-cyclohepta[*l*]phenanthrene-5,8-dione.

(6) 15a-Methyl-2,3,4,5,6,7,8,9,9a,9b,15a,15b-dodecahydro-1*H*-benzo[*b*]cyclohepta[*l*]phenanthrene-10,15-dione.

(7) 2,4a-Dimethyl-4a,4b,5,6,7,8,9,10,11,12,13,14,14a,14b-tetradecahydrodicyclohepta[*a,c*]naphthalene-1,4-dione.

(8) 10b-Methyl-1,2,3,4,5,6,7,8,9,10,10a,10b,16a,16b-tetradecahydrodicyclohept[*a,c*]anthracene-11,16-dione.

(9) 1-Hydroxy-3,20-dimethylpentacyclo[$10.8.0.0^{5,20}.0^{6,12}.0^{13,19}$]icosa-2,18-dien-4-one.

(10) 1,4-Dimethylhexacyclo[$10.8.0.0^{2,11}.0^{4,19}.0^{5,11}.0^{12,18}$]icosane-3,20-dione.

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